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IS 10246 (1982): N-N Diethyl-p-phenylenediamine sulphate, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]



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IS : 10246 - 1982

Indian Standard
SPECIFICATION FOR
N, N-DIETHYL-*p*-PHENYLENEDIAMINE
SULPHATE, PHOTOGRAPHIC GRADE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR N, N-DIETHYL-*p*-PHENYLENE DIAMINE SULPHATE, PHOTOGRAPHIC GRADE

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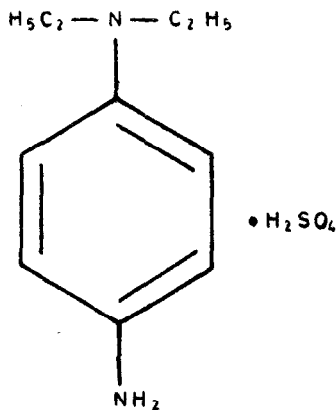
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Indian Standard
SPECIFICATION FOR
N, N-DIETHYL-*p*-PHENYLENE DIAMINE
SULPHATE, PHOTOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1982, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 N, N-diethyl-*p*-phenylenediamine sulphate is used as a photographic colour developer. It is also known by trade names like CD-1, Purechrome-1 and commercial name like TSS chemical. It has the following structural formula:



(Molecular Weight = 262.33)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for N, N-diethyl-*p*-phenylenediamine sulphate used as the basic constituent of developing solutions for treatment of multilayer light sensitive materials of colour photography and cinematography.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to light tan, free flowing crystals, free from aggregates or large crystals and shall consist essentially of N, N-diethyl-*p*-phenylenediamine sulphate.

2.2 The material shall comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR N, N-DIETHYL-*p*-PHENYLENEDIAMINE SULPHATE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Free base, percent by mass	62.5 ± 0.5	A-2
ii)	Ash, percent by mass, <i>Max</i>	0.10	A-3
iii)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	A-4
iv)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	A-5
v)	Volatile matter at 70°C, percent by mass, <i>Max</i>	0.5	A-6
vi)	pH of 5 percent aqueous solution	2.0 ± 0.2	A-7
vii)	Sulphuric acid, percent by mass	37.5 ± 0.5	A-8

3. PACKING, MARKING AND SAFETY PRECAUTIONS

3.1 Packing — The material shall be packed in suitable, dark coloured airtight bottles or as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and legibly and indelibly marked with the following information:

- Name and grade (photographic) of the material;
- Mass of the material;
- Date of manufacture;
- Manufacturer's name and/or his recognized trade-mark, if any; and
- Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification mark may be granted to manufacturers or producers, may be obtained from the Indian Standards Institution.

3.3 Safety Precautions — N, N-diethyl-*p*-phenylenediamine sulphate has a pronounced general toxic action and shows particular effect on liver and central nervous system. On skin contact, it may cause eczema.

3.3.1 Work with N, N-diethyl-*p*-phenylenediamine sulphate should be done only with protective clothing. The place should be provided with exhaust and ventilation systems.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(Clause 2.2 and Table 1)

METHODS OF TEST FOR N, N-DIETHYL-*p*-PHENYLENEDIAMINE SULPHATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2. DETERMINATION OF FREE BASE

A-2.0 General — Using standard ceric sulphate solution, the free base content is found out by volumetric method.

*Specification for water for general laboratory use (*second revision*).

A-2.1 Reagents

A-2.1.1 Dilute Sulphuric Acid — 1 N.

A-2.1.2 Dilute Hydrochloric Acid — 3 N.

A-2.1.3 Ferroin Indicator — see **3.37.1** of IS : 2263-1979*.

A-2.1.4 Standard Ceric Sulphate Solution — 0.05 N.

A-2.2 Procedure — Weigh accurately about 2.5 g of the sample on a watch glass and transfer into a 500-ml volumetric flask and dissolve it in dilute sulphuric acid. Make up to the mark with dilute sulphuric acid.

A-2.2.1 In a one-litre conical flask, take 100 ml of dilute hydrochloric acid and 200 ml of water and add 10 to 12 drops of ferroin indicator. Titrate with ceric sulphate solution to a pale blue end point. Record the blank titre value V_1 . Pipette 50 ml of the sample solution prepared in **A-2.2** into the conical flask and continue titration without refilling the burette until a loss of orange colour is observed momentarily. Reduce the rate of addition as end point is approached, so that one drop is added every 3 to 4 seconds near the end point. The end point is of a greenish yellow colour which does not change with the addition of one more drop.

A-2.3 Calculation

$$\text{Free base, percent by mass} = (V - V_1) \times F \times \frac{0.082 \times 500 \times 100}{20 \times 50 \times M}$$

where

V = nett titre value in ml,

V_1 = blank titre value in ml,

F = factor of 0.05 N ceric sulphate solution, and

M = mass in g of the sample taken for the test.

A-3. DETERMINATION OF ASH

A-3.1 Apparatus

A-3.1.1 Silica Crucible — provided with lid.

A-3.1.2 Furnace — preferably with automatic temperature control device.

A-3.1.3 Desiccator — see IS : 6128-1971†.

*Methods of preparation of indicator solutions for volumetric analysis (first revision).

†Specification for desiccators.

A-3.2 Procedure — Weigh accurately 5.000 g of the material and transfer to a tared silica crucible and then ignite at dull red heat (approximately 600°C). Cool in a desiccator and weigh to get constant mass. Preserve the residue for the determination of iron and heavy metals in **A-4** and **A-5**.

A-3.3 Calculation

$$\text{Ash, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the material taken for the test.

A-4. DETERMINATION OF IRON

A-4.0 General — Two methods have been prescribed for the determination of iron, namely (a) *o*-phenanthroline method and (b) thioglycollic acid method. However, in case of dispute, the *o*-phenanthroline method shall be the referee method.

A-4.1 *o*-Phenanthroline Method

A-4.1.0 General — *o*-phenanthroline, hydroxylamine hydrochloride solution and acetate buffer solution of pH 5 in equal quantities are mixed with the material and the colour is compared with that produced by standard iron solution under identical conditions.

A-4.1.1 Reagents

A-4.1.1.1 Acetate buffer, pH 5 — Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to one litre with water. Adjust the final pH of the solution to 5 ± 0.1 with acetic acid or 10 percent sodium hydroxide solution.

A-4.1.1.2 Hydroxylamine hydrochloride — 10 percent aqueous solution (*m/v*).

A-4.1.1.3 *o*-phenanthroline (1 : 10 - phenanthroline) — 0.1 percent solution (*m/v*).

A-4.1.1.4 Standard iron solution — Dissolve 7.02 g of crystalline ferrous ammonium sulphate [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4) \cdot 6\text{H}_2\text{O}$] in about 300 ml of water containing 2 ml of concentrated sulphuric acid and dilute to 1 000 ml with water. Further dilute one millilitre to 1 000 ml freshly when required. One millilitre of the diluted solution contains 0.001 mg of iron (as Fe).

A-4.1.2 Procedure

A-4.1.2.1 Add 5 ml of concentrated hydrochloric acid to the residue obtained from the determination of ash (*see* **A-3**) and evaporate to dryness. Dissolve the residue in 15 ml of dilute hydrochloric acid and warm on a steam-bath for 10 minutes. Cool, dilute to 50 ml with water in a volumetric flask and mix thoroughly. Use 20 ml of this solution for the determination of iron and preserve the remaining solution for heavy metals test.

A-4.1.2.2 Thoroughly mix equal parts of *o*-phenanthroline, hydroxylamine hydrochloride and acetate buffer of pH 5. Add 5 ml of this mixture to 20 ml portions of both the standard iron solution and the sample solution. Dilute to 50 ml with water and mix well.

A-4.1.2.3 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-4.2 Thioglycollic Acid Method

A-4.2.0 General — Add concentrated hydrochloric acid to the residue obtained from the determination of ash, evaporate to dryness and finally dissolve in dilute hydrochloric acid. Add thioglycollic acid, make the solution alkaline and compare the colour produced with that produced by standard iron solution under identical conditions.

A-4.2.1 Apparatus

A-4.2.1.1 *Nessler cylinders* — 50 ml capacity.

A-4.2.2 Reagents

A-4.2.2.1 *Dilute hydrochloric acid* — 10 percent (*v/v*).

A-4.2.2.2 *Thioglycollic acid* — 89 percent purity, *Min.*

A-4.2.2.3 *Dilute ammonium hydroxide* — 1 : 2.

A-4.2.2.4 *Citric acid solution* — 25 percent (*m/v*).

A-4.2.3 Procedure

A-4.2.3.1 Transfer a 20 ml aliquot of the sample solution from **A-4.1.2.1** to a Nessler cylinder and dilute to about 30 ml. Into the second Nessler cylinder containing 5 ml of dilute hydrochloric acid, pipette 20 ml of standard iron solution and dilute to about 30 ml with water. To both cylinders, add 2 ml of citric acid solution and 2 to 3 drops of thioglycollic acid. Mix, make just alkaline with ammonium hydroxide and dilute to 50 ml.

A-4.2.3.2 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-5. DETERMINATION OF HEAVY METALS

A-5.0 General — The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-5.1 Reagents

A-5.1.1 Dilute Ammonium Hydroxide — 10 percent (*m/m*).

A-5.1.2 Dilute Hydrochloric Acid — approximately 0.1 N.

A-5.1.3 *p*-Nitrophenol Indicator — 0.25 percent aqueous solution (*m/v*).

A-5.1.4 Hydrogen Sulphide — saturated aqueous solution.

A-5.1.5 Standard Lead Solution — Dissolve 0.64 g of lead nitrate in water; add one millilitre of concentrated nitric acid and dilute to 1 000 ml with water. Further dilute 10 to 1 000 ml freshly when required. One millilitre of the resulting solution contains 0.004 mg of Pb.

A-5.2 Procedure

A-5.2.1 To the 20 ml portion of the sample solution (see **A-4.1.2.1**) in a Nessler cylinder add a few drops of *p*-nitrophenol indicator and then add ammonium hydroxide dropwise, until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless again and then add 0.5 ml in excess. Add 5 ml of hydrogen sulphide solution and dilute to 50 ml.

A-5.2.2 At the same time transfer 5 ml of standard lead solution into another Nessler cylinder and render it neutral to *p*-nitrophenol indicator by using dilute ammonium hydroxide and dilute hydrochloric acid as described in **A-5.2.1**. Add 0.5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml.

A-5.2.3 The material shall be considered to have passed the requirement of the test if the colour produced in the sample solution is not more intense than that produced in the standard solution.

A-6. DETERMINATION OF VOLATILE MATTER

A-6.0 General — A known quantity of the material is dried at 70°C and loss in mass is taken as volatile matter.

A-6.1 Apparatus

A-6.1.1 *Glass-Stoppered Shallow Weighing Bottle*

A-6.1.2 *Oven* — preferably with automatic temperature control device.

A-6.1.3 *Desiccator* — see IS : 6128-1971*.

A-6.2 Procedure — Weigh accurately 3 ± 0.1 g of the material in the weighing bottle and dry in the oven at 70°C for about 8 hours. Cool in the desiccator and weigh.

A-6.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M - M_1) \times 100}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material obtained on drying.

A-7. pH OF THE SOLUTION

A-7.0 General — The pH of a 5 percent solution of the material is determined by using a suitable pH meter.

A-7.1 Apparatus

A-7.1.1 *pH Meter* — with glass and calomel electrodes.

A-7.2 Procedure — Prepare a five percent solution of the material in a 100-ml beaker, using freshly boiled and cooled water. Measure the pH of the solution using a pH meter.

A-8. DETERMINATION OF SULPHURIC ACID

A-8.0 General — Sulphates are precipitated using barium chloride and determined gravimetrically. From this, the sulphuric acid content is found out.

A-8.1 Reagents

A-8.1.1 *Barium Chloride Solution* — approximately 10 percent (m/v).

*Specification of desiccators.

A-8.1.2 Concentrated Hydrochloric Acid

A-8.2 Procedure — Weigh accurately 5 ± 0.1 g of the sample, transfer it to the 100 ml volumetric flask, dissolve and make up to the mark using water. Pipette 20 ml of this made up solution into a 250-ml beaker containing 80 ml water and one millilitre of hydrochloric acid and heat to boil. To this hot solution, add about 10 ml of hot barium chloride solution and continue stirring at boiling temperature for 2 minutes to complete the precipitation of barium sulphate. Allow to heat on a water bath for 1–2 hours and filter through Whatman No. 40 filter paper. Wash the precipitate by hot water until the filtrate is free from chloride. Incinerate and ignite the precipitate in a tared silica crucible to constant mass.

A-8.3 Calculation

$$\text{Sulphuric acid, percent by mass} = M \times 0.4202 \times \frac{100}{20} \times \frac{100}{M_1}$$

where

M = mass in g of the precipitate, and

M_1 = mass in g of the material taken for the test.

APPENDIX B

(Clause 4.1)

**SAMPLING OF N, N-DIETHYL-*p*-PHENYLENEDIAMINE
SULPHATE, PHOTOGRAPHIC GRADE**

B-1. GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions shall be observed.

B-1.2 Samples shall not be taken at a place exposed to weather.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw representative samples, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and airtight glass or plastic containers.

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B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lot.

B-2.2 From each lot five containers shall be selected (*see B-2.2.1*) for drawing samples for test. In case the lot contains less than five containers, each container shall be tested.

B-2.2.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed. In case this standard is not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and, starting from any one, count them as 1, 2, , r , where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every r th container thus counted shall be withdrawn to constitute the sample.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 Drawing of Samples — Draw a representative sample from each container selected.

B-3.1.1 Mix portions of the material drawn from the same container to prepare the test sample representing the container. The quantity of material to be drawn from each container shall be sufficient to conduct all the tests given in 2 and shall be not less than 200 g.

B-3.1.2 The samples shall be stored in dry glass bottles or other suitable containers, sealed airtight and labelled with all the particulars of sampling given in B-1.7.

*Methods for random sampling.

B-3.1.3 Five test samples, representing the five containers selected as in **B-2.2** shall constitute a set of test samples.

B-3.2 Out of the portions of material from all the five containers selected, approximately equal quantities of the material shall be taken and mixed together to form a composite sample of quantity sufficient to carry out triplicate determinations for each of the characteristics tested on the composite sample.

B-3.2.1 The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third to be used as a referee sample.

B-3.3 The remaining portions of the material from each of the five containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples, representing the container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual and composite samples shall be immediately transferred to separate sample containers and labelled with full identification particulars.

B-3.5 The referee samples consisting of a composite sample and a set of individual samples, shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of free base, heavy metals and iron shall be conducted on each of the five individual samples. Tests for all other characteristics prescribed in 2 and Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 Tests Carried Out on Five Individual Test Samples

B-5.1.1 *Free Base* — From the test results for free base, the mean (\bar{X}) and range (R) of test results shall be calculated (range being defined as the difference between the maximum and the minimum values of test results). The lot shall be considered to have passed in respect of assay, if the value of the expression ($\bar{X} \pm 0.6 R$) lies between 62.0 and 63.0.

B-5.1.2 *Heavy Metals and Iron* — The lot shall be considered to have passed for the characteristics tested if each test sample passes the corresponding test prescribed.

B-5.2 Test Carried Out on Composite Test Sample — The lot shall be considered to have passed in respect of the characteristics tests on the composite test sample (*see* **B-4.1**) if the composite test sample satisfies the corresponding test prescribed.

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2**.